

**CLAIMS**

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- 5        1. A colloidal dispersion, in an organic solvent, of microfibrils and/or microcrystals of a fibrillar organic substance selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta 1 \rightarrow 3$  glucan,  $\beta 1 \rightarrow 3$  xylan and  $\beta 1 \rightarrow 4$  mannan, containing in addition at least one compound possessing a hydrophilic part and a hydrophobic part.
- 10      2. A dispersion according to Claim 1, characterized in that the compound possessing a hydrophilic part and a hydrophobic part is selected from the group comprising a surfactant, a stabilizing polymer, a co-surfactant or mixtures thereof, and especially a mixture of surfactant and co-surfactant.
- 15      3. A dispersion according to Claim 1 or Claim 2 of microfibrils and/or microcrystals of cellulose.
- 20      4. A dispersion according to any one of the Claims 1 to 3, characterized in that the organic solvent has a dielectric constant that is less than or equal to approx. 37.5, and/or in that the organic solvent is selected from the group comprising:
- 25      – aliphatic hydrocarbons having from about 5 to about 20 carbon atoms, especially pentane, hexane, heptane, octane, dodecane or cyclohexane,
- 30      – aromatic hydrocarbons, especially xylene, toluene or decalin,
- 35      – chlorine-containing solvents, especially chloroform, dichloromethane, carbon tetrachloride, dichloroethane,
- 40      – ketones having from about 3 to about 10 carbon atoms, especially acetone or methyl ethyl ketone,
- 45      – polymerizable vinylic compounds, especially methacrylates and acrylates of alkyls having from about 1 to about 10 carbon atoms, styrene, vinyl acetate,
- 50      – epoxides, especially those with an aliphatic chain having from about 1 to about 10 carbon atoms, and/or an aromatic chain, the diepoxides, the triepoxides and/or the tetraepoxides,

- primary, secondary or tertiary amines, especially those with an aliphatic chain having from about 1 to about 10 carbon atoms and/or an aromatic chain, the diamines, triamines or tetramines,
- alkyl acetates having from about 1 to about 10 carbon atoms, especially methyl, ethyl, propyl or butyl acetates,
- ethers with an alkyl chain having from about 1 to about 20 carbon atoms or an aromatic chain such as ethyl ether, or benzyl ether,
- aldehydes, carboxylic acids and/or their acylated derivatives and anhydrides, the polyacids, with an alkyl chain having from about 1 to about 20 carbon atoms or an aromatic chain such as acetaldehyde, acetic acid, maleic anhydride, benzaldehyde,
- primary, secondary or tertiary alcohols, especially those with an aliphatic chain having from about 1 to about 10 carbon atoms, and/or an aromatic chain, the polyalcohols, especially methanol, ethanol, isopropanol, butanol or benzyl alcohol,
- tetrahydrofuran (THF), pyridine, dimethylformamide (DMF), dimethylacetamide (DMAc),
- mineral and/or organic oils, of synthetic or natural origin, such as silicone oils or vegetable oils,
- or mixtures thereof.

5. A dispersion according to any one of the Claims 1 to 4, characterized in that the quantity of cellulose varies from about 0.01 wt.% to about 50 wt.% relative to the total weight of the dispersion.

25 6. A dispersion according to any one of the Claims 1 to 5, characterized in that the compound possessing a hydrophilic part and a hydrophobic part is:

(a) a surfactant possessing:

- a hydrophilic part, which is capable of being adsorbed on the microfibrils and/or microcrystals of the compound selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta$  1  $\rightarrow$  3 glucan,  $\beta$  1  $\rightarrow$  3 xylan and  $\beta$  1  $\rightarrow$  4 mannan, and containing for example oxyethylene groups,
- a hydrophobic part, containing for example a carbon chain having at least 6 carbon atoms, aromatic or non-aromatic, and capable of interacting with the solvent, the said surfactant being selected in particular from the group comprising:

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- \* cationic surfactants, for example from the family of the quaternary ammonium alkyls containing from about 1 to about 2 alkyl substituents, having from about 6 to about 20 carbon atoms, such as didecyldimethyl ammonium bromide,
  - \* anionic surfactants, for example from the family of the polyoxyalkylenated alkarylphenol phosphoric esters, in which the alkyl substituent contains from about 1 to about 12 carbon atoms, and from about 5 to about 25 oxyalkylene units having from about 1 to about 4 carbon atoms, and especially oxyethylene, oxypropylene or oxybutylene units, for example BNA, a mixture of ester and diester of phosphoric acid with an alkaryl chain, in which the alkyl substituent contains 9 carbon atoms and 9 oxyethylene units,
  - \* amphoteric surfactants possessing a quaternary ammonium group and an anionic phosphoric group, for example from the family of the phospholipids, such as egg or soya lecithin,
  - \* neutral surfactants, for example from the family of those containing a sorbitol unit, and one to about 3 polyoxyethylene chains, one to about 3 fatty chains having from about 12 to about 30 carbon atoms, and especially 18 carbon atoms, such as polyoxyethylene (20) sorbitan trioleate,

(b) or, a stabilizing polymer possessing from about 5 to about 200 hydrophilic units and from about 10 to about 200 hydrophobic units, especially those with a molecular weight below 20000, such as ethylene oxide-propylene oxide block copolymers.

7. A dispersion according to any one of the Claims 1 to 6, characterized in that the co-surfactant possesses:

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- a hydrophilic part that is compatible with the hydrophilic part of the compound possessing a hydrophilic part and a hydrophobic part as defined in Claim 6, and especially with the hydrophilic part of the surfactant or of the stabilizing polymer as defined in Claim 6,
  - a hydrophobic part that is compatible with the hydrophobic part of the compound possessing a hydrophilic part and a hydrophobic part as defined in Claim 6, and especially with the hydrophobic part of the surfactant or of the stabilizing polymer as defined above,

the co-surfactant making it possible, moreover, for the microfibrils and/or microcrystals of the fibrillar organic substance as defined in Claim 1 to be rendered compatible with the organic solvent,

5           the said co-surfactant being selected in particular from the group comprising alcohols having from about 4 to about 18 carbon atoms, carboxylic acids having from about 4 to about 18 carbon atoms, aldehydes having from about 4 to about 18 carbon atoms or amines having from about 4 to about 18 carbon atoms.

10           8. A dispersion according to any one of the Claims 1 to 7, characterized in that it contains:

- cellulose microfibrils and/or microcrystals, in a quantity varying from about 0.01 wt.% to about 50 wt.% relative to the total weight of the dispersion, and especially from about 0.1 wt.% to about 30 wt.%,
- an organic solvent as defined in Claim 4, in a quantity varying from about 50 wt.% to about 99.9 wt.% relative to the total weight of the dispersion,
- a surfactant as defined in Claim 6, in a quantity varying from about 0.01 wt.% to about 50 wt.% relative to the total weight of the dispersion,
- and if necessary a co-surfactant as defined in Claim 7, in a quantity varying from about 0 wt.% to about 20 wt.% relative to the total weight of the dispersion.

20           9. A dispersion according to any one of the Claims 1 to 8, characterized in that it exhibits at least one of the following properties:

- it does not form aggregates (it is non-flocculent),
- it is birefringent in shear,
- it is stable for periods ranging from at least one minute to at least 12 months.

25           10. A method of preparation of a dispersion according to any one of the Claims 1 to 9, characterized in that:

30           (1) an aqueous dispersion of microfibrils and/or microcrystals of a fibrillar organic substance selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta$  1 → 3 glucan,  $\beta$  1 → 3 xylan and  $\beta$  1 → 4 mannan, is mixed with a compound possessing a hydrophilic part and a hydrophobic part selected from the group comprising a surfactant, a stabilizing polymer, a co-surfactant or mixtures

thereof, the said surfactant and the said stabilizing polymer being as defined in Claim 6, and the said co-surfactant being as defined in Claim 7,

to obtain an aqueous dispersion of microfibrils and/or of microcrystals of the aforesaid fibrillar organic substance as defined according to any one of the Claims 10 to 13,

(2) the water is eliminated from the aqueous dispersion as obtained in the preceding stage to obtain a dry mixture of surfactant and/or of stabilizing polymer and possibly of co-surfactant, and a fibrillar organic substance selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta$  1  $\rightarrow$  3 glucan,  $\beta$  1  $\rightarrow$  3 xylan and  $\beta$  1  $\rightarrow$  4 mannan,

(3) the mixture as obtained in the preceding stage is dispersed in an organic solvent until a dispersion is obtained.

**11. A method of preparation according to Claim 10, characterized in that:**

(1) an aqueous dispersion of microfibrils and/or of microcrystals of cellulose is mixed with a surfactant selected from the group comprising BNA, polyoxyethylene sorbitan trioleate or didecyldimethyl ammonium bromide,

the weight ratio between (a) the said surfactant and (b) the said microfibrils and/or microcrystals of cellulose varying from about 0.1:1 to about 20:1, to obtain an aqueous colloidal dispersion of microfibrils and/or of microcrystals of cellulose as defined according to any one of the Claims 9 to 12,

(2) the water is eliminated from the aqueous dispersion as obtained in the preceding stage to obtain a dry mixture of surfactant and cellulose, the said mixture containing from about 5 wt.% to about 95 wt.% of surfactant relative to the total weight of the mixture, and from about 5 wt.% to about 95 wt.% of cellulose relative to the total weight of the mixture,

(3) the mixture as obtained in the preceding stage is dispersed in an organic solvent as defined in Claim 4,

until a dispersion of cellulose microfibrils and/or microcrystals is obtained for which the percentage by weight of adsorption between the said surfactant and the said cellulose microfibrils and/or microcrystals varies from about 0.1 to about 20, and especially from about 0.1 to about 5.

12. Use of a dispersion according to any one of the Claims 1 to 9, for the preparation of gels, liquid crystals or materials containing cellulose microfibrils and/or microcrystals.

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5 13. An organic solvent as defined in Claim 4, thickened and/or viscous, characterized in that it contains:

- All*  
10 – microcrystals and/or microfibrils of a fibrillar organic substance selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta 1 \rightarrow 3$  glucan,  $\beta 1 \rightarrow 3$  xylan and  $\beta 1 \rightarrow 4$  mannan,  
– a compound possessing a hydrophilic part and a hydrophobic part selected from the group comprising a surfactant, a stabilizing polymer, a co-surfactant or their mixtures, the said surfactant, and stabilizing polymer being as defined in Claim 6 and the said co-surfactant being as defined in Claim 7.

15 14. A dry mixture of surfactant and/or of stabilizing polymer and possibly of co-surfactant, and of a fibrillar organic substance selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta 1 \rightarrow 3$  glucan,  $\beta 1 \rightarrow 3$  xylan and  $\beta 1 \rightarrow 4$  mannan.

20 15. A method of preparation of a dry mixture according to Claim 14, characterized in that

25 (1) an aqueous dispersion of microfibrils and/or microcrystals of a fibrillar organic substance selected from the group comprising cellulose, chitin, and polysaccharides such as  $\beta 1 \rightarrow 3$  glucan,  $\beta 1 \rightarrow 3$  xylan and  $\beta 1 \rightarrow 4$  mannan, is mixed with a compound possessing a hydrophilic part and a hydrophobic part selected from the group comprising a surfactant, a stabilizing polymer, a co-surfactant or mixtures thereof, the said surfactant and the said stabilizing polymer being as defined in Claim 6, and the said co-surfactant being as defined in Claim 7,

30 to obtain an aqueous dispersion of microfibrils and/or microcrystals of the aforesaid fibrillar organic substance as defined according to any one of Claims 10 to 13,

(2) the water is eliminated from the aqueous dispersion as obtained in the preceding stage to obtain the aforesaid mixture.

16. Use of a dry mixture according to Claim 14, for the preparation of materials containing microfibrils and/or microcrystals of cellulose.

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